

NP10 8QQ

REC'D 2 1 AUG 2003

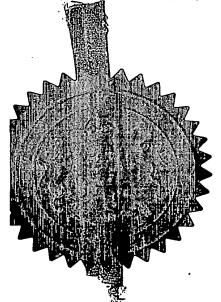
**WIPO** PCT

I, the undersigned, being an officer duly authorised in accordance with Section 74(1) and (4) of the Deregulation & Contracting Out Act 1994, to sign and issue certificates on behalf of the Comptroller-General, hereby certify that annexed hereto is a true copy of the documents as originally filed in connection with the patent application identified therein.

In accordance with the Patents (Companies Re-registration) Rules 1982, if a company named in this certificate and any accompanying documents has re-registered under the Companies Act 1980 with the same name as that with which it was registered immediately before reregistration save for the substitution as, or inclusion as, the last part of the name of the words "public limited company" or their equivalents in Welsh, references to the name of the company in this certificate and any accompanying documents shall be treated as references to the name with which it is so re-registered.

In accordance with the rules, the words "public limited company" may be replaced by p.l.c., plc, P.L.C. or PLC.

Re-registration under the Companies Act does not constitute a new legal entity but merely subjects the company to certain additional company law rules.



## PRIORITY DOCUMENT

SUBMITTED OR TRANSMITTED IN COMPLIANCE WITH

Signed #

11 August 2003 Dated

**BEST AVAILABLE COPY** 

An Executive Agency of the Department of Trade and Industry

24JUL02 E735487-2-00 P01/7700 0.00-021 17077.7 The Patent Office

Cardiff Road Newport Gwent NP9 1RH

68007103001

2 3 JUL 2002

Fee: £0

McLean

BARRIER

LONDON

91001

WC1X 8PL

NSM/JEM/44844.GB01

0217077.7

Mars, Incorporated

Virginia 22101-3883

United States of America

6885 Elm Street

Your reference

you fill in this form.)

Patent application number

(The Patent Office will fill in this part)

Full name, address and postcode of the or of each applicant (underline all surnames)

Request for grant of a patent

an explanatory leaflet from the fatent office to help "C;

Patents ADP number (if you know it)

If the applicant is a corporate body, give the

country/state of incorporation

Title of the invention

Full name, address and postcode in the United Kingdom to which all correspondence relating to this form and translation should be sent

Patents ADP number (if you know it)

If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and (if you know it) the or each application number

If this application is divided or otherwise

derived from an earlier UK application, give the number and the filing date of

Country

Reddie & Grose

16 Theobalds Road

Priority application (If you know it)

Date of filing (day/month/year)

Number of earlier application

Date of filing (day/month/year)

Is a statement of inventorship and of right to grant of a patent required in support of this request? (Answer 'Yes' if:

the earlier application

a) any applicant named in part 3 is not an inventor, or

b) there is an inventor who is not named as an applicant, or

c) any named applicant is a corporate body. See note (d))

YES

Patents Form 1/77

#### Patents Form 1/77

Described the number of sheets for any of the cowing items you are filing with this form. Do not count copies of the same document.

#### Continuation sheets of this form

Description

10 ~

Claim(s)

3/

Abstract

Zan'

Drawing(s)

1 4

10. If you are also filing any of the following, state how many against each item.

Priority documents

Translations of priority documents

Statement of inventorship and right to grant of a patent (Patents Form 7/77)

Request for preliminary examination and search (Patents Form 9/77)

Request for substantive examination (Patents Form 10/77)

Any other documents (please specify)

ið

11.

I/We request the grant of a patent on the basis of this application.

Signature

Date

Reddie & Grase

23 407.02

12. Name and daytime telephone number of person to contact in the United Kingdom

N S MARLOW 020-7242 0901

#### Warning

After an application for a patent has been filed, the Comptroller of the Patent Office will consider whether publication or communication of the invention should be prohibited or restricted under Section 22 of the Patents Act 1977. You will be informed if it is necessary to prohibit or restrict your invention in this way. Furthermore, if you live in the United Kingdom, Section 23 of the Patents Act 1977 stops you from applying for a patent abroad without first getting written permission from the Patent Office unless an application has been filed at least 6 weeks beforehand in the United Kingdom for a patent for the same invention and either no direction prohibiting publication or communication has been given, or such direction has been revoked.

#### Notes

- a) If you need help to fill in this form or you have any questions, please contact the Patent Office on 0645 500505.
- b) Write your answers in capital letters using black ink or you may type them.
- c) If there is not enough space for all the relevant details on any part of this form, please continue on a separate sheet of paper and write "see continuation sheet" in the relevant part(s). Any continuation sheet should be attached to this form.
- d) If you have answered 'Yes' Patents Form 7/77 will need to be filed.
- e) Once you have filled in the form you must remember to sign and date it.

#### BARRIER

The present invention relates to novel barrier layers for products such as foodstuffs and to methods of making the novel barrier layers.

5

10

15

20

25

30

The ingress of oxygen and migration of moisture, fat and/or other components in foodstuffs can often severely limit their shelf life. Such processes are difficult to suppress and can lead to oxidation, softening, blooming and invidious changes in taste, texture, smell and appearance of foodstuffs. The application of edible barrier layers, films or coatings to foodstuffs to obviate such problems is known in the art. These layers, films and coatings are intended to prevent, or at least reduce, either the ingress of oxygen or moisture from the atmosphere into a foodstuff or the migration of moisture, fat and/or other components between different layers or phases within a foodstuff. Typically, in foodstuffs where fat migration is likely to be an issue, rather than employing a barrier layer to obviate the problem, fat migration is avoided by manipulation of the ingredients of the foodstuff to omit or reduce the levels of those fats prone to migration.

Many known barrier layers, films or coatings suffer, however, from the disadvantages that they are difficult to apply, expensive or have an adverse effect on the taste or texture of foodstuffs to which they are applied. Furthermore, in order for known layers, films and coatings to be effective as barriers on foodstuffs it is often necessary to apply them in a thick layer, which exaggerates any detrimental effect upon the taste or texture they may have.

According to the invention there is provided a solid barrier layer formed by the application of a molten polyol,

polyol derivative or mixture thereof to at least part of the surface of a substrate.

The present invention allows an edible, thin, continuous solid barrier layer to be applied to a substrate. The invention mainly finds application in foodstuffs by application of polyols to edible substrates, but also has wider applications by application of polyols to non-edible substrates.

Preferably, the thickness of the barrier layer is about 1.0mm or less, more preferably about 0.5mm or less.

5

15

20

Preferably, the barrier layer is at least 80% continuous, more preferably at least 95% continuous.

Where the barrier layer is a barrier to moisture migration, preferably the barrier layer is formed by application of a polyol having a solubility at  $25^{\circ}$ C of less than about 50g/100g H<sub>2</sub>O, more preferably less than 30g/100g H<sub>2</sub>O.

Where the barrier layer is a barrier to moisture migration, preferably the polyol is mannitol, erythritol or isomalt, most preferably mannitol.

The barrier layer may be formed from a single molten polyol or polyol derivative or from a molten mixture of two or more polyols and/or polyol derivatives.

The barrier layer may also comprise other common additives and ingredients such as colourants, flavourants, acidulants and plasticisers provided that the layer still provides an effective barrier against the ingress of oxygen or migration of moisture, fat and/or other components.

Also according to the invention there is provided a method of forming a solid barrier layer on at least part of the surface of a substrate comprising: melting a polyol, polyol derivative or mixture thereof; applying the polyol, polyol derivative or mixture thereof to a surface of the substrate; and solidifying the molten polyol, polyol derivative or mixture thereof to form a barrier layer.

Preferably, the barrier layer is formed by dipping the substrate in the molten polyol, polyol derivative or mixture thereof, by spraying, painting or otherwise applying the molten polyol, polyol derivative or mixture thereof to the substrate or by enamelling molten polyol, polyol derivative or mixture thereof on the substrate.

10

20

25

30

Where the barrier layer is a barrier to moisture migration, preferably, the polyol is mannitol, erythritol or isomalt, most preferably mannitol.

The hard, close packing crystalline or amorphous glass nature of barrier layers according to the invention makes them effective as protection against oxygen ingress and flavour and odour transfer.

Since the barrier layers produced according to the invention are odourless, colourless and clear or translucent, the invention, also enables barriers to be applied to foodstuffs that do not adversely affect the smell or appearance of the foodstuffs.

The thin barrier layers produced according to the invention are crisp/crunchy. The invention, therefore, also enables barrier layers to be applied to foodstuffs, such as breadsticks, croutons or fresh fruit and vegetables, which advantageously enhance the crisp/crunchy texture of the foodstuffs.

A number of polyols, in particular mannitol, erythritol and isomalt, have a low degree of hygroscopicity and low solubility in water. By application of a thin layer of such polyols to substrates, the method of the invention enables barriers to be formed which provide effective and lasting protection against moisture migration. The invention also enables other polyols and/or polyol derivatives, such as maltitol, lactitol, sorbitol, xylitol and isomaltulose, having low solubilities in water at temperatures below 0°C, to be used to form barrier layers effective against moisture migration in frozen foodstuffs, for example on biscuits in ice cream.

5

10

15

20

25

30

Applying a thin, for example 0.5mm or less, layer of a molten polyol, polyol derivative or mixture thereof to substrates by the method of the invention produces an edible thin, solid, continuous barrier which has improved mechanical strength over for example known fat-based moisture barriers. By reducing the thickness of the layer required to produce an effective barrier, the present invention enables a barrier to be applied to foodstuffs which does not adversely affect the consumer acceptance of the foodstuffs.

The polyol barrier layers according to the present invention also exhibit improved temperature stability over known fat-based moisture barriers. Most fats liquify above 40°C and higher melting waxes impart an unpleasant waxy mouth feel to foodstuffs or other substrates to which they are applied. In contrast to most fats, polyols melt at high temperatures, typically above 100°C, and the water solubility of, for example, mannitol increases only slightly with temperature.

The insolubility of polyols in fats and oils makes them suitable for use as barriers against fat migration. Through the formation of solid polyol barrier layers providing effective protection against fat migration, the present

invention allows the production of multi-phase or component foodstuffs containing fats which are prone to migration.

The low viscosity of molten polyols enables solid barrier layers according to the invention to be formed by application of polyols to substrates by a wide variety of known techniques, such as dipping, spraying and enamelling. The low viscosity of molten polyols also aids their distribution over the surface of substrates. Consequently, solid barrier layers according to the invention are smooth and uniform.

The invention will be further described, by way of the following examples of specific embodiments thereof:

# Example 1

5

10

15

25

at 170°C. Each flake was submerged in the molten mannitol before being removed and any excess polyol shaken off. The flakes were then allowed to cool. Cold skimmed milk was then poured onto the dipped cornflakes and also onto eight equally sized portions of untreated cornflakes.

### 20 Example 2

from Example 1 and the mixture reheated to 170°C. Whole, shortbread biscuits, halved shortbread biscuits and quartered shortbread biscuits were then dipped halfway into the molten mannitol, before being removed and allowed to cool. Once cool, the other halves of the shortbread biscuits were dipped in the molten mannitol and the biscuits then removed once again and allowed to cool. The biscuits were then floated in cold skimmed milk for 10 minutes.

#### Example 3

5

10

15

20

25

30

Shortbread biscuits were dipped in molten mannitol as in Example 2 and the biscuits allowed to cool. Once cool, approximately 15 holes were punched in the surface of the biscuits and half of each biscuit then submerged in hot coffee.

Example 1 produced cornflakes with a hard, continuous crystalline polyol layer over their entire surface. . polyol layer resulted in no change in the taste or sweetness of the cornflakes, but did result in a slightly crunchier texture compared to the untreated cornflakes. minutes in cold milk, the crunchiness of the untreated cornflakes began to deteriorate and after 4 minutes in cold milk the untreated cornflakes were no longer crunchy and very In contrast, after 20 minutes in cold milk the soggy. cornflakes with the polyol layer remained as crunchy as the untreated cornflakes were at the time the milk was added. After 25 minutes in cold milk, the crunchiness of the cornflakes with the polyol layer began to deteriorate, resembling that of the untreated cornflakes after 3 minutes in milk.

Example 2 produced biscuits with a hard, continuous crystalline polyol layer over their entire surface. The polyol layer resulted in no change in the taste or sweetness of the biscuits, but did result in a slightly crunchier texture. After 10 minutes floating in milk, no moisture penetration of the polyol layer was observed.

Example 3 produced biscuits with a hard, continuous crystalline polyol layer over their entire surface as in Example 2. When the biscuits were dipped in hot coffee a darkening of the biscuits beneath the barrier layer was observed, indicating the absorption of coffee into the

biscuits through the holes punched through the barrier layer. The barrier layer, however, remained intact.

Although in the above Examples the substrates were coated using molten mannitol, the same techniques may be employed to coat substrates with molten erythritol, isomalt or other polyols, polyol derivatives or mixture thereof. It will be appreciated that if molten erythritol or isomalt are used instead of molten mannitol a lower temperature may be employed due to the lower melting points of erythritol and isomalt compared to mannitol, about 124°C and 147°C respectively as compared to 167°C.

5

10

15

20

25

30

In all of the above Examples the substrates were dipped into the molten polyol in order to form the barrier layer. It will be readily appreciated that a variety of other techniques, such as spraying and enamelling, wherein the polyol is applied to the substrate as a powder and heat then applied to melt the polyol, could alternatively be employed.

While cornflakes and shortbread biscuits are employed as substrates in the Examples given above, it will be readily appreciated by those skilled in the art that barrier layers according to the invention may be applied to a wide variety of other edible and non-edible substrates. It will also be appreciated that while in Examples 1 and 2 the barrier layers are employed to reduce moisture migration, barrier layers according to the invention could be alternatively employed to reduce fat migration, oxygen ingress or odour or flavour transfer. In addition, because of their high mechanical strength and the ease with which they can be applied, it will be appreciated that barrier layers according to the invention may also be employed to prevent melt deformation or, as in Example 3, deformation due to moisture absorption.

It will also be appreciated that while in the above Examples a solid barrier layer was applied to the entire

surface area of the substrate, barrier layers according to the invention may be applied to only part of the surface of a substrate. This may be desirable where for example only one surface of a substrate having low water activity is exposed to a high water activity phase. For example, a barrier layer according to the invention may be applied to one surface of a pizza base in order to prevent moisture migration into the base from toppings subsequently applied to that surface.

The application of a solid barrier layer according to the invention to only part of the surface area of a substrate will be further illustrated with reference to the accompanying drawings in which:

15

Figure 1 shows a side elevation of a layered foodstuff comprising a barrier layer according to the invention.

Figure 2 shows a cross-sectional view of a paper plate having a barrier layer according to the invention applied to its upper surface; and

Figure 1 shows a layered foodstuff 10 comprising a biscuit layer 11 having a lower surface 12 and an upper surface 13, 20 and a caramel layer 14 on top of the biscuit layer 11, having a lower surface 15, adjacent the upper surface 13 of the biscuit layer 11, and an upper surface 16. A barrier layer 17 according to the invention is applied to the upper surface 13 of the biscuit layer 11, which would otherwise be exposed 25 to the lower surface 15 of the caramel layer 14. The barrier layer 17 reduces migration of moisture from the caramel layer 14 into the biscuit layer 11, thereby preventing the biscuit layer 11 from becoming soft and the caramel layer 14 from The crisp, crunchy nature of the becoming dry and hard. 30 barrier layer 17 also enhances the crunchiness of the It will be appreciated that barrier

according to the invention could also be applied to the lower surface 12 of the biscuit layer 13 and/or upper surface 16 of the caramel layer 14 if desired, for example to prevent moisture ingress from the atmosphere into the biscuit if the lower surface 12 of the biscuit layer is exposed to the air.

5

10

15

20

25

Barrier layers according to the invention may, be similarly applied for example confectionery products having a coconut creme layer to reduce migration of fat from the coconut into the remainder of the confectionery, for example to prevent discolouration of a chocolate layer which would otherwise be in contact with the coconut.

Figure 2 shows a paper plate 20 having a lower surface 22 and an upper surface 24 which is exposed to foodstuffs during use of the plate. A barrier layer 26 according to the invention is applied to the upper exposed surface 24 of the plate 20 to reduce migration into the plate of moisture, fat and other components from foodstuffs placed on the upper surface 24, thereby preventing mechanical failure of the plate.

Barrier layers according to the invention may be similarly applied for example to the internal surfaces of paper cups to reduce migration into the cups of moisture from beverages contained therein and so prevent mechanical failure of the cups. The edible, colourless, clear or translucent and biodegradable nature of barrier layers according to the invention makes them particularly suitable for this purpose.

Examples of possible substrates to which barrier layers according to the invention could be advantageously applied include:

breakfast cereals;

pizza bases;

biscuit in biscuit and caramel confectionery products; bread or pastry in prepared sandwiches, wraps and baked goods;

paper plates;

5

25

croutons; and

dried fruit.

# To reduce fat migration:

coconut creme or fat based centres in chocolate confectionery products;

paper plates;

wafers in wafer products with cream fillings; and chocolate products to reduce soft fat migration driven bloom.

To reduce oxygen ingress or odour/flavour transfer:

nuts; and

different flavoured foodstuffs packaged in the same container.

# To reduce deformation;

20 chocolate bars/blocks to resist melt deformation in hot climates;

iced confections to resist melt deformation during nonchilled transport or storage; and

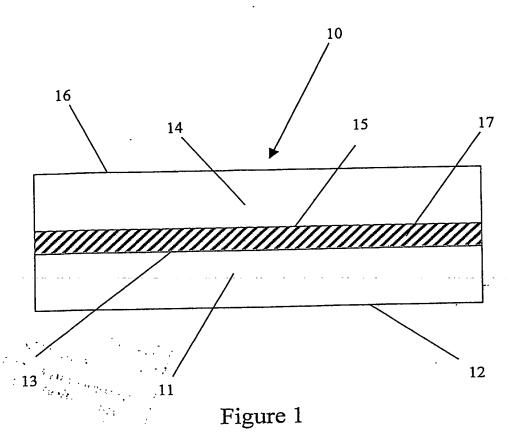
biscuits to resist deformation when dunked in hot or cold beverages.

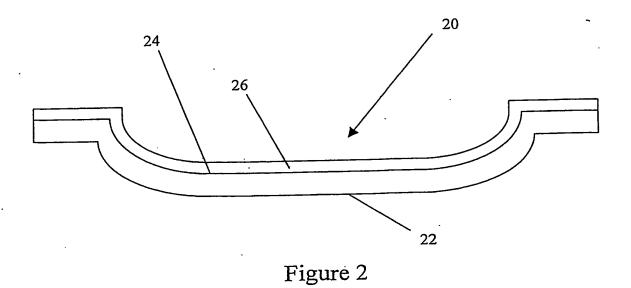
#### CLÁIMS

- 1. A solid barrier layer formed by the application of a molten polyol, polyol derivative or mixture thereof to at least part of the surface of a substrate.
- 5 2. A barrier layer according to claim 1 having a thickness of 1.0mm or less.
  - 3. A barrier layer according to claim 1 or 2 having a thickness of 0.5mm or less.
- 4. A barrier layer according to any preceding claim which is at least 80% continuous.
  - 5. A barrier layer according to any preceding claim which is at least 95% continuous.
  - 6. A barrier layer that reduces the migration of fat according to any preceding claim.
- 7. A barrier layer that reduces the ingress of oxygen or flavour or odour transfer according to any preceding claim.
  - 8. A barrier layer that reduces deformation of the substrate according to any preceding claim.
- A barrier layer that reduces the migration of moisture
  according to any preceding claim.
  - 10. A barrier layer according to claim 9 formed by application of a polyol having a solubility at 25°C of less than  $50g/100g\ H_2O$ .

- 11. A barrier layer according to claim 9 or 10 formed by application of a polyol having a solubility at  $25^{\circ}$ C of less than  $30g/100g \text{ H}_2\text{O}$ .
- 12., A barrier layer according to any of claims 9 to 11 formed by application of mannitol, erythritol or isomalt.
  - 13. A product comprising a barrier layer according to any preceding claim.
  - 14. A foodstuff according to claim 13.
- 15. Use of a molten polyol, polyol derivative or mixture thereof to form a solid barrier layer.
  - 16. Use according to claim 15 of a polyol having a solubility at 25°C of less than  $50g/100g\ H_2O$ .
  - 17. Use according to claim 15 or 16 of a polyol having a solubility at 25°C of less than  $30g/100g\ H_2O$ .
- 18. Use according to any of claims 15 to 17 of mannitol, erythritol or isomalt.
  - 19. A method of forming a solid barrier layer on at least part of the surface of a substrate comprising:
- (a) melting a molten polyol, polyol derivative or 20 mixture thereof;
  - (b) applying the polyol, polyol derivative or mixture thereof to a surface of the substrate; and
  - (c) solidifying the molten polyol, polyol derivative or mixture thereof to form a barrier layer.
- 20. A method according to claim 19 wherein the barrier layer is formed by spraying the molten polyol, polyol derivative or mixture thereof onto the substrate.

- 21. A method according to claim 19 wherein the barrier layer is formed by dipping the substrate in the molten polyol, polyol derivative or mixture thereof.
- 22. A method according to claim 19 wherein the barrier layer is formed by enamelling of the polyol, polyol derivative or mixture thereof.
  - 23. A method of forming a barrier layer that reduces the migration of fat according to any of claims 19 to 22.
- 24. A method of forming a barrier layer that reduces the ingress of oxygen or flavour or odour transfer according to any of claims 19 to 23.
  - 25. A method of forming a barrier layer that reduces deformation of the substrate according to any of claims 19 to 24.
- 26. A method of forming a barrier layer that reduces the migration of moisture according to any of claims 19 to 25.
  - 27. A method according to claim 26 wherein the polyol is mannitol, erythritol or isomalt.
- 28. A method of forming a barrier layer on a foodstuff according to any of claims to 19 to 27.
  - 29. A solid barrier layer substantially as described with reference to the examples.
    - 30. Use of a molten polyol substantially as described with reference to the examples.
- 25 31. A method of forming a solid barrier layer substantially as described with reference to the examples.





# This Page is Inserted by IFW Indexing and Scanning Operations and is not part of the Official Record.

# **BEST AVAILABLE IMAGES**

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:
BLACK BORDERS
☐ IMAGE CUT OFF AT TOP, BOTTOM OR SIDES
☐ FADED TEXT OR DRAWING
☐ BLURRED OR ILLEGIBLE TEXT OR DRAWING
☐ SKEWED/SLANTED IMAGES
☐ COLOR OR BLACK AND WHITE PHOTOGRAPHS
☐ GRAY SCALE DOCUMENTS
☐ LINES OR MARKS ON ORIGINAL DOCUMENT
☐ REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY
Потикр.

# IMAGES ARE BEST AVAILABLE COPY.

As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.